

Real-Time Characterization of Hot-Wire CVD Growth of Si:H Films Using Spectroscopic Ellipsometry

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ABSTRACT

This paper reports on the application of real-time spectroscopic ellipsometry (RTSE) to in-situ characterization of hot-wire chemical vapor deposition (HWCVD) of Si:H films. We have investigated the evolution of film morphology and crystallinity as a function of hydrogen dilution $R=[H_2]/[SiH_4]$ for films grown at a substrate temperature of 200°C. RTSE shows that the films undergo a roughening transition at a thickness of approximately 170 Å, independent of R . We have correlated the RTSE measurements with AFM measurements of surface roughness and Raman scattering measurements of crystallinity.

1. Introduction

RTSE has been successfully applied to characterize the microstructural and phase evolution of hydrogenated Si (Si:H) films prepared by low-temperature RF plasma-enhanced chemical vapor deposition (PECVD) [1]. In that work, the authors showed that the surface roughness, as characterized by RTSE, is a sensitive indicator of changes in the phase and microstructure of the growing films. The degree of crystallinity is clearly indicated by the dielectric function as measured by RTSE.

2. Experimental

The thin-film silicon samples were grown by HWCVD on c-Si substrates [2]. The reactor was configured with a single tungsten filament that was heated to ~2,200°C. The silane flow was fixed at 20 sccm whereas the hydrogen flow was varied between 0 and 400 sccm, causing the dilution R to vary from 0 to 20. The throttling valve was fixed for each run at 57% open, causing the chamber pressure to increase with increasing hydrogen flow. The substrate temperature was ~200°C at the start of the deposition.

RTSE spectra were collected over the range of 300 to 1500 nm at 70° angle of incidence using a rotating compensator ellipsometer (Woollam M2000). Spectra were collected four times per second during the first ~200 Å of growth and every two seconds thereafter.

The Raman scattering measurements were performed in a 180° backscattering configuration with a 50-mW air-cooled Ar-ion laser operating at 488.1 nm and a single-grating Spex 270M spectrometer with a LN₂-cooled CCD array detector. A holographic notch filter was used to suppress the laser line. AFM measurements were performed using an Autoprobe LF from ThermoMicroscopes with silicon cantilevers. The tip radius was 100 Å.

3. Results and Discussion

Spectroscopic ellipsometry measures the quantities ψ and δ , which relate to changes in the relative amplitude and phase of the two polarization components of light reflected from the sample's surface. These measured quantities are translated into more physically meaningful quantities through model-based analysis, wherein a mathematical model of the sample is used to calculate theoretical values of ψ and δ . These theoretical values are then compared with the experimentally measured ones, and the model parameters are adjusted until an optimal fit to the experimental data is obtained. With the proper software and a sufficiently powerful computer, this analysis can be performed in real time during the sample growth.

The Si:H thin films in this study were modeled as a single "bulk" layer of a-Si:H on top of a layer of native SiO₂ on a crystalline-Si substrate. Also included in the model is a surface roughness layer, modeled as an effective medium consisting of a 50/50 mix of the underlying a-Si:H and voids. Model parameters adjusted during fitting included the surface roughness thickness, the bulk a-Si:H thickness, and the Si substrate temperature. The dielectric function of the a-Si:H bulk layer was adjusted in order to achieve the best global fit over all measured times.

Figure 1 shows the evolution of surface roughness and bulk thickness for a dilution level of $R=6$.

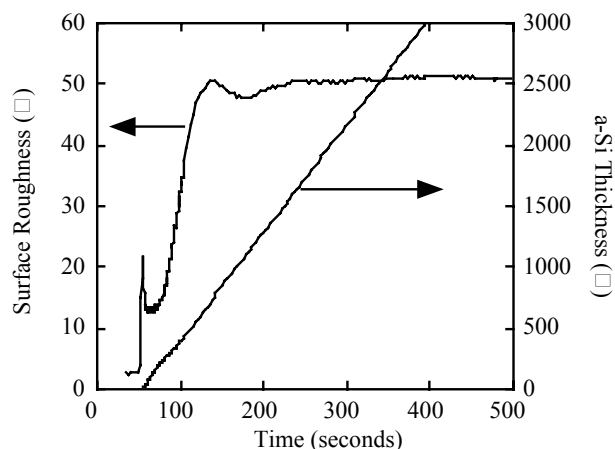


Fig 1. Surface roughness and bulk layer thickness for the first 500 seconds of deposition for $R=6$.

The early-time behavior seen in the figure is typical of all depositions studied. Silane gas is introduced into the chamber, and the shutter between the filament and the substrate is opened at approximately 50 seconds. There is a rapid increase in surface roughness from near zero to greater than 20 Å. This value then rapidly drops to about 13 Å

before beginning a gradual ascent toward its maximum. The initial rise in surface roughness is due to nucleation of separate islands of a-Si:H on the crystalline-Si substrate. These islands reach a height greater than 20 Å before the islands begin to coalesce into a bulk film. The coalescence of the islands produces a smoothening effect, which reduces the surface roughness to ~13 Å.

Figure 2 shows a plot of surface roughness vs. bulk thickness for varying hydrogen dilution. The initial nucleation stage is not shown because the bulk thickness remains zero during that phase. Following initial nucleation, there is a general trend of surface smoothening until approximately 170 Å thickness. At this point the films with dilutions of R=0, 1, and 3 begin gradually roughening and reach a final roughness of ~25 Å. The dielectric functions that provided the best fit to the real-time data are consistent with amorphous silicon optical properties.

We have used AFM to characterize the surface roughness after the films are removed from the growth chamber. For the R=0 and R=1 films, we have excellent agreement. The RTSE and AFM values are within ± 2 Å of each other. For the higher dilution films, RTSE measures surface roughness values 10–30 Å greater than those measured by AFM. We have noted that the surface roughness decreases somewhat during the first few minutes after the film growth has been terminated, but have not followed this evolution for the duration of the cool-down period. It is possible that further smoothening occurs for the high-dilution films. It is also possible that the close agreement for the low-dilution films is fortuitous, and that differences in measured values of surface roughness are due to differing definitions of surface roughness for AFM vs. RTSE. Further study is needed.

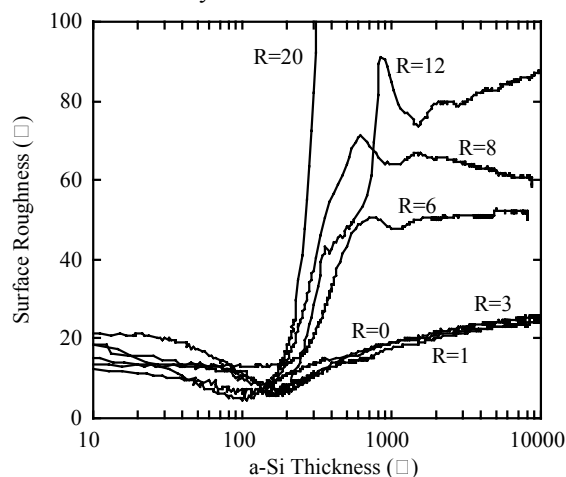


Fig 2. Surface roughness plotted against bulk thickness for different hydrogen dilution levels R=0, 1, 3, 6, 8, 12, and 20.

For films with dilution of R=6 and greater, there is also a roughening transition at ~170 Å bulk thickness. For these higher levels of dilution, the roughening is much more severe than at lower dilution. For R=6, 8, and 12 the roughness eventually levels off and even decreases somewhat, indicating a second smoothening transition. For R=20, the roughness increases beyond 200 Å before leveling off, again with a second smoothening transition, not

shown on the graph in Figure 2. According to the work of Collins et al. on PECVD growth of Si:H films, a rapid increase in surface roughness followed by a second smoothening transition is indicative of microcrystalline-Si nucleation and bulk layer formation.

In contrast to that work, our RTSE measurements find no evidence of microcrystalline Si in our films. We have verified these results using Raman scattering to probe the crystallinity of the films. The results are shown in Figure 3 below. Crystalline Si has a strong, sharp Raman peak at ~ 510 cm⁻¹. There is no evidence of such a peak in any of the spectra. The broad Raman peak at ~ 480 cm⁻¹ is consistent with a-Si:H. Hence, we conclude that there is no microcrystalline Si present within the sensitivity level of Raman scattering or spectroscopic ellipsometry.

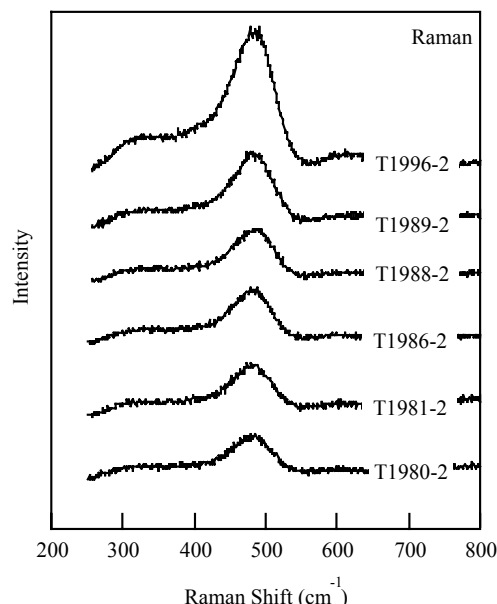


Fig 3. Raman spectra for all of the Si:H films in this study.

4. Conclusions

Our RTSE measurements of HWCVD growth of Si:H thin films have demonstrated behavior similar to that found by Collins and coworkers for PECVD growth of Si:H films. We find a rapid initial increase in surface roughness, indicating nucleation of the film. This is followed by gradual smoothening, then further roughening transitions. In contrast to Collins' work, we do not find evidence for amorphous-to-microcrystalline transitions. We anticipate that amorphous-to-microcrystalline transitions will be found at lower silane flow rates.

5. Acknowledgements

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